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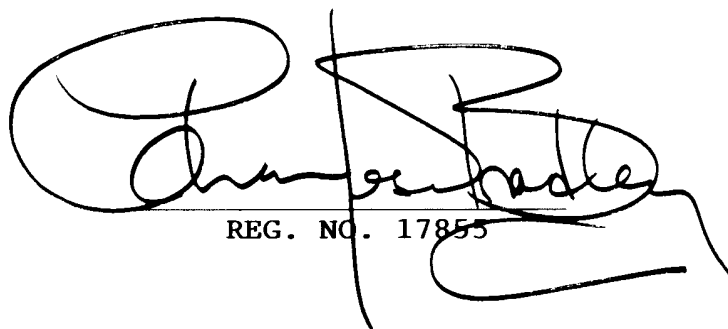
In re: Reissue Patent Application of )  
Inventors: TOMOHIRO KAWASE et al. ) Group Art: 1765  
Serial No.: 09/824,965 ) Examiner: Robert Kunemund  
Filed: April 3, 2001 )  
Title: Method Of Preparing Group III-V )  
Compound Semiconductor Crystal )

Director of Technology Group 1765  
Assistant Commissioner for Patents  
Washington, D.C. 20231

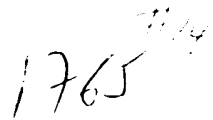
**PROOF OF SERVICE**

I hereby certify that on September 24, 2002, I served the foregoing PROTEST  
UNDER 37 C.F.R. 1.291 and the cited Marshall-DeCuir article upon counsel of record for  
Kawase, by causing two true and correct copies thereof to be served by hand delivery on the  
following:

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REG. NO. 17855



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## I. INTRODUCTION

As required by 37 C.F.R. § 1.291(b), this protest includes: (a) a listing of the patents, publications, or other information relied upon (Form 1449 attached), (b) a concise explanations of the relevance of each listed item, and (c) a copy of each listed patent or publication or other item.

## II. THE PRIOR ART ARTICLE RELIED ON AND ITS RELEVANCE

**Reference AV:** H.D. Marshall & D.W. DeCuir; "A novel technique to reduce the concentration of carbon in LEC gallium arsenide"; pages 960 – 962 of Journal of Crystal Growth 110 (1991) North Holland.

The above Marshall-DeCuir article concerns reducing the concentration of carbon in gallium arsenide and then adding carbon in a controlled fashion, in order to control the amount of carbon and consequently the amount and distribution of the carbon in the resulting GaAs ingot, and its consequent semi-insulating properties.

The explanation in the article of how the initial carbon level is reduced is not particularly germane here. What is germane is how the carbon level is thereafter brought up to the desired level. Nevertheless, we note that the carbon level is reduced to a level below that ultimately desired by bubbling  $\text{As}_2\text{O}_3$  vapor through the GaAs melt, as shown in Fig. 1. In this regard, the article states in the left hand column on page 962 that:

The  $\text{As}_2\text{O}_3$  injection technique reduced the carbon concentration to a level below that desired for semi-insulating electrical properties.

Having reduced the carbon to a low level by the use of  $\text{As}_2\text{O}_3$  vapor, which results in a crystal designated "C", shown in table 1 of the article, the desired carbon level is then obtained by placing in a crucible carbon fiber,  $\text{B}_2\text{O}_3$ , and crystal C (a GaAs compound). In this regard, the article states:

To restore these properties, crystal C was carbon doped and regrown. Carbon doping was accomplished by vigorously rubbing 0.0012 g of 99.99% carbon fiber into the inside bottom of the PBN

crucible prior to loading with GaAs. The resulting crystal D contained significantly more carbon than C and had the desired electrical properties as shown in table 1.

Table 1 in the article lists the carbon content and insulating properties of four crystals, designated crystals A, B, C and D, each constituting a GaAs compound. Crystal C was formed by creating a melt from crystals A and B and then reducing the carbon content by bubbling  $\text{As}_2\text{O}_3$  vapor, as explained. Crystal D was then prepared by adding carbon, using carbon fiber, to the GaAs crystal C in the presence of  $\text{B}_2\text{O}_3$ . As can be seen from table 1, the carbon, and consequent semi-insulating properties, are relatively uniform in the resulting crystal D.

### **III. THE MARSHALL-DeCUIR ARTICLE ANTICIPATES KAWASE PATENT CLAIMS 1, 2, 4, 6, 9, 13, 18, 19, 20, 21 AND 22**

It will be seen from the above discussion that the Marshall-DeCuir article anticipates each of the Kawase '622 patent claims 1, 2, 4, 6, 9, 13, 18, 19, 20, 21 and 22. A claim chart applying each limitation of each of these claims follows.

### **IV. CLAIM CHART APPLYING KAWASE CLAIMS AT ISSUE TO THE MARSHALL-DeCUIR ARTICLE**

#### **Claims of Reissue Application.**

1. A method of preparing a carbon-doped group III-V compound semiconductor crystal, comprising the steps of:

placing a compound raw material, solid carbon, and a boron oxide substance into a crucible or a boat,

#### **Reference AV – Marshall-DeCuir**

A method of preparing a carbon-doped group III-V compound semiconductor crystal, namely a GaAs crystal. P. 962, left column.

Placing the following enumerated items into the crucible illustrated in Fig. 1 on page 961.

- solid carbon comprised of 0.0012 grams carbon fiber (p. 962);
- "crystal C", which is a compound GaAs material of about 4 kg. Crystal "C" was prepared from GaAs crystals A and B as described on page 962; and

## Claims of Reissue Application.

## Reference AV – Marshall-DeCuir

- boric oxide (page 960, bottom of right column) is the encapsulant. It is illustrated as  $B_2O_3$  in Fig. 1 on page 961. 1,200 ppmw of wet basic oxide is used.

sealing said crucible or boat containing said compound raw material, said solid carbon, and said boron oxide substance within an airtight vessel formed of a gas impermeable material,

Sealing the crucible of Fig. 1 containing the compound GaAs material (crystal C), the fiber carbon, and the boric oxide in the airtight vessel (the pull chamber of Fig. 1, page 961) formed of a gas impermeable material. Page 960, right column. The last paragraph refers to an argon ambient of 30 psi gauge pressure.

heating and melting said compound raw material in said crucible or said boat sealed within said airtight vessel, and

The GaAs compound (crystal C) is heated and melted in the crucible sealed within the airtight vessel in Fig. 1. It is melted at 30 degrees C above the seed-in temperature (melt in), allowing the melt temperature to stabilize for 30 minutes. Page 961, middle of left column through middle of right column.

solidifying said melted compound raw material to grow a carbon-doped compound semiconductor crystal,

The melt temperature is reduced to seed-in temperature, and seeding is begun 2 hours later. The growth rates is constant at 8.5mm/h. Page 961, right column.

wherein an amount of said solid carbon placed into said crucible or said boat is larger than an amount of carbon doped into said compound semiconductor crystal.

The "doping potential" of the solid carbon (1.2 mg) placed into crucible with 4 kg of GaAs is more than 20 times larger than the carbon concentrations doped into the compound, namely,  $3.3E+15$  and  $3.7E+15$ , measured respectively at the shoulder and tail.<sup>1</sup>

<sup>1</sup> With regard to the limitation in claim 1 that the amount of carbon placed into the crucible is larger than that doped into the crystal, as shown in the Marshall-DeCuir articles, this is an inherent characteristic of carbon doping since the process of doping solid carbon into a crystal is less than 100% efficient.

*(Continued on next page)*

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The Kawase '622 patent states in the Summary Of The Invention that the weight of the carbon used is preferable greater than that doped into the crystal, preferably at least ten times greater, and preferably at least 100 times greater. (col. 3, lines 37-39, 44-49)

The examples do not indicate the actual weight of the carbon in the crystals, and in fact the weight of the carbon in a crystal is so much less than the weight of the GaAs that it is not possible to weigh it. For example, in Kawase Example 2, using the ratios of the carbon doping in the table below (i.e., 166 and 154, or an average of 160), the amount of carbon in the crystal is about  $0.04 \text{ g} \div 160 = 0.00025 \text{ grams}$ , while the weight of the GaAs is 10,000 grams.

One measure can, however, be used. It is the "doping potential," designated "y." A standard equation for the doping potential is as follows:

$$y = 6.023 \times \frac{\text{Carbon (in ppm)}}{1E + 6} \times \frac{5.36 \text{ GaAs (g/cm}^3\text{)}}{12 \text{ C (g/mole)}} \times 1E + 23 \text{ (atoms/mole)}$$

From the tables and text in the Kawase patent and the table in the Marshall-DeCuir article, the value of y can be calculated for the Kawase Examples 1 to 4 and Marshall-De Cuir crystal D. Also, the ratio of y to the carbon content measured at the Shoulder of the crystal and at the Tail can be calculated.

The following table shows the values specified in the Kawase and Marshall-DeCuir documents and the calculated values:

|   |                      |                        |                | Carbon<br>doping<br>potential<br>"Y" | CARBON MEASURED<br>atoms/cm3 |                       | Ratio of calculated<br>carbon doping<br>potential to carbon<br>concentration<br>measured in ingot |             |
|---|----------------------|------------------------|----------------|--------------------------------------|------------------------------|-----------------------|---|-------------|
|   | (1) C<br>(grams)     | (2)<br>GaAs<br>(grams) | (3) C<br>(ppm) | (4)(atoms<br>C/cm3)                  | (5)<br>Shoulder              | (6) Tail              | (7)<br>Shoulder   | (8)<br>Tail |
| EXAMPLE 1-<br>C powder                    | 0.100 <sup>1</sup>   | 3,000 <sup>1</sup>     | 33.3           | 8.96E+18                             | 1.4E+15 <sup>2</sup>         | 8.0E+14 <sup>2</sup>  | 6,400   | 11,200      |
| EXAMPLE 2- C fiber                        | 0.040 <sup>3</sup>   | 10000 <sup>3</sup>     | 4.00           | 1.08E+18                             | 6.5E+15 <sup>4</sup>         | 7.0E+15 <sup>4</sup>  | 166   | 154         |
| EXAMPLE 3- C fiber                        | 0.020 <sup>5</sup>   | 10000 <sup>5</sup>     | 2.00           | 5.38E+17                             | 2.3E+15 <sup>8</sup>         | 2.2E+15 <sup>8</sup>  | 234   | 245         |
| EXAMPLE 4- C fiber                        | 0.0075 <sup>7</sup>  | 10000 <sup>7</sup>     | 0.75           | 2.02E+17                             | 1.3E+15 <sup>8</sup>         | 1.2E+15 <sup>8</sup>  | 155   | 168         |
| REFERENCE AV <sup>11</sup><br>CRYSTAL "D" | 0.0012 <sup>10</sup> | 4000 <sup>11</sup>     | 0.30           | 8.07E+16                             | 3.3E+15 <sup>12</sup>        | 3.7E+15 <sup>12</sup> | 24.46   | 21.81       |

<sup>1</sup> From '622 table 1; <sup>2</sup> From '622 table 2; <sup>3</sup> From '622 table 3; <sup>4</sup> From '622 table 4; <sup>5</sup> From '622 col. 8, lines 14-18; <sup>6</sup> From '622 table 5; <sup>7</sup> From '622, col. 8, lines 34-37; <sup>8</sup> From '622 table 6; <sup>10</sup> From Marshall-DeCuir article, p. 962, left col.; <sup>11</sup> From M-D article, pp. 960-961; <sup>12</sup> From M-D article, p. 961, table 1.

(Continued on next page)

## Claims of Reissue Application.

## Reference AV – Marshall-DeCuir

2. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 1, further comprising a step of heating and melting said boron oxide substance and having said melted boron oxide substance in contact with at least a portion of said solid carbon, during said step of heating and melting said compound raw material.

The  $B_2O_3$  flows freely toward the bottom of the crucible at about  $500^\circ C$ , far below the melting point of GaAs (about  $1,238^\circ C$ ), and thus contacts the carbon fiber.<sup>2</sup>

4. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 1, wherein said boron oxide substance comprises boron oxide and water.

A wet basic oxide (1,200 ppmw) was used as the encapsulant. Page 960, bottom of right column.

6. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 1, wherein said amount of said solid carbon placed into said crucible or said boat is at least 10

As shown by the table in footnote 1, the ratio is 24.46 to 1 at the shoulder and 21.81 to 1 at the tail.

*(Continued from previous page)*

The calculated values listed in the above table are shown bold. A sample calculation is as follows, using Example 1:

$$y = 6.023 \times \frac{33.3}{1E + 6} \times \frac{5.36}{12} \times 1E + 23 = 8.96E + 18$$

$$\text{Ratio}_{\text{Shoulder}} = \frac{\text{col. 4}}{\text{col. 5}} = \frac{8.96E + 18}{1.40E + 15} = 6,400$$

$$\text{Ratio}_{\text{Tail}} = \frac{\text{col. 4}}{\text{col. 6}} = \frac{8.96E + 18}{8.40E + 14} = 11,200$$

It will be seen that all of the ratio values of carbon, applied to the carbon measured at the shoulder and tail, all exceed the value of 10. Unless this method of calculating the ratio is employed, there is no support for the claim limitations in the Kawase examples.

No calculation has been made for Kawase Example 5, which used a carbon disc, since the weight of the disc is not specified in the '622 patent. It is clear, however, that the entire disc was not incorporated into the crystal.

<sup>2</sup> See Bourret-Courchesne patent 5,131,975, col. 3, lines 47-51.

## Claims of Reissue Application.

## Reference AV – Marshall-DeCuir

times larger than said amount of carbon doped into said compound semiconductor crystal.

9. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 1, further comprising a step of maintaining said melted compound raw material in a melted state for a certain time period before said step of solidifying said melted raw material to grow said crystal.

13. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 1, wherein said solid carbon comprises fiber carbon.

18. A method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 1, wherein said crucible or said boat comprises pBN.

19. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 1, wherein said compound raw material comprises GaAs, and wherein said compound semiconductor crystal comprises a GaAs crystal.

20. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 2, further comprising having said melted boron oxide substance in contact with at least a portion of said melted compound raw material, during said step of heating

The melt is allowed to stabilize for 30 minutes, before reducing the melt temperature to seed in temperature, and seeding is begun 2 hours later. Growth rates were constant at 8.5 mm/h. Therefore the hold time is 2.5 hours. Page 961, middle of left column through middle of right column.

Carbon doping was accomplished by vigorously rubbing 0.0012 g of 99.99% carbon fiber into the inside bottom of the PBN crucible prior to loading with GaAs. Page 962, left column, middle last full paragraph.

The crucible comprises PBN. Page 962, left column, middle last full paragraph.

The raw material is a GaAs crystal "C", while grown crystal is a GaAs crystal "D".

As shown in Fig. 1, B<sub>2</sub>O<sub>3</sub> lays on the GaAs melt. Page 961.

**Claims of Reissue Application.**

**Reference AV – Marshall-DeCuir**

and melting said compound raw material.

**V. CONCLUSION**

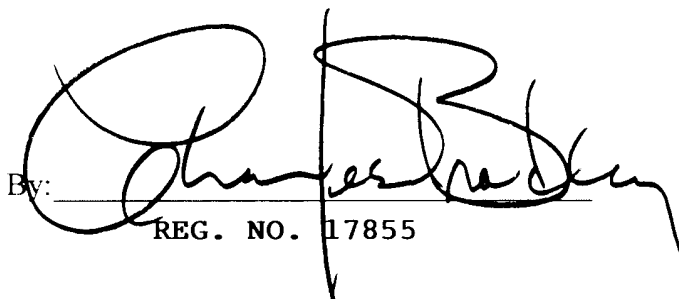
This protest demonstrates that claims 1, 2, 4, 6, 9, 13, 18, 19, 20, 21, and 22 are anticipated by the teachings of Reference AV: H.D. Marshall & D.W. DeCuir; "A novel technique to reduce the concentration of carbon in LEC gallium arsenide"; pages 960 – 962 of Journal of Crystal Growth 110 (1991) North Holland. It demonstrates that Marshall-DeCuir produced crystal "D" of the article by the steps enumerated in claim 1, and that the requirements of the "wherein" clause in claim 1 are met during growth of crystal "D". Specifically, the ratio of the doping potential of the of carbon fiber placed into the crucible by Marshall-DeCuir to grow crystal "D" is 24.46 or 21.81 times larger than the resulting carbon concentrations at the Shoulder and the Tail.<sup>3</sup> As such, each and every element of the above claims is present in Reference AV, as indicated in the foregoing claim chart.

Respectfully submitted,

Date: \_\_\_\_\_

9/24/02

By: \_\_\_\_\_

  
REG. NO. 17855

<sup>3</sup> Note that one form of solid carbon in the Kawase '622 patent is carbon fiber. ('622 patent, col. 4, lines 50-51)